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# The Effects of Cigarette Filter Ventilation on Delivery and Retention of Organic Acids *

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SUMMARY

A method for simultaneous identification and quantitative determination of 30 organic acids was established. The smoke yields and filter retentions of organic acids and routine smoke components, total particulate matter (TPM), nicotine-free dry particulate matter (NFDPM), nicotine and carbon monoxide (CO) at different filter ventilation levels were determined under both International Organization for Standardization (ISO) and Health Canadian Intense (HCI) smoking regimes. As a result of smoke dilution during filter ventilation, the yields of all organic acids were reduced in mainstream cigarette smoke. The spatial distribution pattern of the concentration of each organic acid in the filter was investigated at different ventilation levels and their filter retention determined. On one hand, the concentration of organic acids with a lower boiling point (BP) and lower molecular weight (MW) was relatively higher at the smoking end and the periphery part of the filter and spatial concentration distributions within the filter were significantly affected by smoke diffusion. On the other hand, those acids with high BPs and high MW were mainly distributed at the tobacco rod end and central part of the filter and spatial concentration distributions were only slightly influenced by their smoke diffusion within the filter whilst air compression around the filter vents also led to less change. This way, different acids in mainstream cigarette smoke were reduced to different extents which can also influence the acid-base equilibrium and sensory quality of the smoke. Compared with ISO smoking regime, the vent blocking and more intense smoking HCI regime led to different extents of yield increase for each of the studied acids. The effect of filter ventilation in the HCI smoking regime was not investigated, as the HCI smoking regime requires blocked ventilation holes. [Contrib. Tob. Nicotine Res. 30 (2021) 199–211]

ZUSAMMENFASSUNG

Es wurde eine Methode zur Identifizierung und gleichzeitigen quantitativen Bestimmung von 30 organischen Säuren entwickelt. Bei unterschiedlichen Filterventilationsgraden wurden entsprechend dem Abrauchprotokoll der Internationalen Organisation für Normung (ISO) und dem Health Canada Intense-Abrauchprotokoll (HCI) die Rauchausbeute und Filterretention von organischen Säuren sowie reguläre Rauchbestandteile, das Gesamtkondensat (TPM), das nikotinfreie Trockenkondensat (NFDPM) und der Gehalt an Nikotin und Kohlenmonoxid (CO) ermittelt. Aufgrund der Rauchverdünnung bei der Filterventilation war die Ausbeute im Hauptstromrauch bei allen organischen Säuren reduziert. Bei unterschiedlichen Ventilationsgraden wurde das räumliche Konzentrationsverteilungs-

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muster jeder organischen Säure im Filter untersucht und deren Filterretention bestimmt. Zum einen war die Konzentration organischer Säuren mit niedrigerem Siedepunkt (BP) und geringerem Molekulargewicht (MG) am Mundende und am Peripherieteil des Filters relativ höher und die räumliche Konzentrationsverteilung im Filter wurde signifikant von der Rauchdiffusion beeinflusst. Zum anderen waren die Säuren mit hohem BP und hohem MG hauptsächlich am Tabakstrangende und am zentralen Teil des Filters verteilt und der Einfluss der Rauchdiffusion im Filter auf die räumliche Konzentrationsverteilung war nur gering. Die Luftverdichtung um die Filteröffnungen führte ebenfalls zu weniger Veränderungen. Dadurch waren die Säuren im Hauptstromrauch in unterschiedlichem Ausmaß reduziert. Dies kann auch einen Einfluss auf das Säure-Base-Gleichgewicht und die sensorische Qualität des Rauchs haben. Im Vergleich zum ISO-Abrauchprotokoll nahm unter dem intensiveren HCI-Abrauchprotokoll mit Blockierung der Lüftungsöffnungen die Ausbeute bei jeder der untersuchten Säuren in unterschiedlichem Maß zu. Für das HCI-Abrauchprotokoll wurde der Effekt der Filterventilation nicht untersucht, da bei diesem Rauchprotokoll eine Blockierung der Filteröffnungen erforderlich ist. [Contrib. Tob. Nicotine Res. 30 (2021) 199–211]

## RESUME

Une méthode servant simultanément à l'identification et à la détermination quantitative de trente acides organiques fut mise au point. Les rendements de fumée et les retentions des filtres pour les acides organiques et les composants habituels de la fumée, la matière particulaire totale (MPT), la matière particulaire anhydre exempte de nicotine, la nicotine et le monoxyde de carbone (CO) à différentes intensités de ventilation du filtre furent mesurés tant selon les paramètres de fumage de l'organisation internationale de normalisation (ISO) que selon ceux de Health Canada Intense (HCI). En raison de la dilution de la fumée due à la ventilation du filtre, il s'avéra que les rendements de tous les acides organiques étaient réduits dans la fumée principale. Le modèle de distribution spatiale de la concentration de chaque acide organique dans le filtre fut analysé à différentes intensités de ventilation et leur rétention au niveau du filtre fut mesurée. D'une part, la concentration des acides organiques présentant un point d'ébullition plus bas et un poids moléculaire (p.m.) plus faible était relativement plus élevée à l'extrémité de tirage et dans la portion périphérique du filtre et les répartitions spatiales des concentrations à l'intérieur du filtre étaient influencées de façon significative par la dispersion de la fumée. D'autre part, ces acides présentant un point d'ébullition plus élevé et un p.m. plus lourd étaient principalement répartis à l'extrémité du boudin de tabac et dans la portion centrale du filtre et les répartitions spatiales des concentrations n'étaient que légèrement affectées par la dispersion de la fumée à l'intérieur du filtre alors que la compression de l'air autour des orifices du filtre contribuait aussi à de moindres changements. Ainsi, il fut observé que différents acides présents dans la fumée principale étaient réduits dans diverses amplitudes, un phénomène aussi susceptible d'influencer l'équilibre acido-basique et la qualité sensorielle de la fumée. Comparativement aux paramètres de fumage ISO, les paramètres HCI prévoyant un fumage plus intense et bloquant les orifices produisirent différentes amplitudes d'augmentation de rendement pour chacun des acides étudiés. L'effet de la ventilation du filtre sur les paramètres de fumage de HCI ne fut pas étudié puisque ce régime de fumage exige le blocage des orifices de ventilation. [Contrib. Tob. Nicotine Res. 30 (2021) 199–211]

## INTRODUCTION

Organic acids are important chemical components in tobacco and cigarette smoke (1–5). The organic acids in cigarette smoke are not only related to the smoke pH, but also associated with aroma quality and irritation (6). Therefore, the analysis of organic acids in tobacco and their yields in cigarette smoke have received great attention (7–8). MOLDOVEANU *et al.* (9) established a LC-MS method for determining 10 organic acids in tobacco and the method showed good sensitivity. MI *et al.* (10) identified 36 organic acids in cigarette smoke and found that 2-propenoic acid and 3-butenoic acid have an obvious influence on aroma and taste of cigarette smoke. In addition, filter ventilation has been widely used in cigarette design and it significantly affects the release of chemical components, including organic acids in cigarette smoke (11–16). BROWNE *et al.* (17) studied the effect of filter ventilation on the distribution of NFDPM, nicotine, water, CO and carbon dioxide in mainstream and sidestream smoke. The results reported that the sidestream component yields were not changed significantly, and the mainstream smoke yields obviously decreased when the cigarette filter was ventilated. It was inferred that smoke dilution by filter ventilation resulted in a decrease in total puff volume and a decrease of tobacco consumed.

However, MORIE (18) noted unexpectedly large reductions in carbon monoxide with ventilated filter cigarettes. NORMAN *et al.* (19) also reported that nicotine and NFDPM gave differential changes in filter retention when filtered cigarettes were ventilated. More recently, XIE *et al.* (20) investigated the relationship between filter ventilation and the release of 25 acidic components in mainstream cigarette smoke and found that different components were decreased in different relative proportions, when the filter ventilation was increased. The research of JING *et al.* (21) showed that the extent of the reduction of flavour component yields at different filter ventilation levels were related to their BPs and MWs. CHEN *et al.* (22) studied the effects of ventilation on the release of seven harmful components in mainstream cigarette smoke, and found that the reduction rates of CO, hydrogen cyanide (HCN) and crotonaldehyde were higher than those of 4-(methylnitrosamino)-1-(3-pyridyl)-1-butanone (NNK) and benzo-[a]-pyrene (B[a]P) when filter ventilation was increased. In fact, it was found that smoke yields were not only affected by smoke dilution during filter ventilation.

However, the reasons for the effect of filter ventilation on components of different chemical classes in cigarette smoke and the associated ventilation effect on sensory quality are still unclear. The purpose of this work was to help clarify this through the investigation of the filter

retention and smoke yields of organic acids in ventilated cigarettes, and to provide data for product design and development.

In the present study, a gas chromatography-mass spectrometry (GC-MS) method for simultaneous identification and quantitative determination of 30 organic acids was used. The effect of filter ventilation on the retention and smoke yields of organic acids and routine smoke components was analyzed under the ISO smoking regime. Furthermore, the spatial concentration distribution of some organic acids, nicotine and water were investigated at different filter ventilation levels, and a possible mechanism for smoke diffusion in ventilated cigarette filters was proposed.

## EXPERIMENTAL

### *Cigarette samples*

Cigarette samples with cellulose acetate (CA) filters were prepared for testing in the experiments. The design specifications are shown in Table 1.

**Table 1. Design specification of cigarette samples**.

| Cigarette description | Value |
|---|---|
| Tobacco rod length (mm) | 57 |
| Filter tip length (mm) | 27 |
| CA filter specification (denier) | 3.0/Y32000 |
| Cigarette circumference (mm) | 24.3 |
| Blend style | Chinese flue-cured tobacco |
| Weight of cigarette (g) | 0.89 |
| Filter ventilation (%) | Pressure drop (Pa) |
| 0±1 | 1301~1399 |
| 10±1 | 1244~1342 |
| 20±1 | 1195~1293 |
| 30±1 | 1036~1134 |
| 40±1 | 942~1040 |

### *Determination of organic acids in mainstream cigarette smoke*

The cigarette samples were smoked on a Cerulean SM450 (Cerulean, Milton Keynes, UK) smoking machine according to the ISO (35 mL puff volume, 2 s puff duration, 1 puff/min) and HCI (55 mL puff volume, 2 s puff duration, 1 puff/30 s) standard conditions. In order to trap organic acids in the gas phase of the mainstream cigarette smoke, one impinger, containing dichloromethane (20 mL) and *trans*-2-hexenoic acid (50 µg/mL) as an internal standard (IS), was connected to the Cambridge filter pad holder. TPM from 5 cigarettes under ISO smoking or 2 cigarettes under HCI smoking was collected on 44-mm Cambridge pads. The content of organic acids in the gas phase and in TPM were determined respectively.

After smoking was completed, an aliquot (1 mL) of the smoke vapour collected in the impinger solution was transferred to the chromatogram vial, and bis(trimethylsilyl)trifluoroacetamide (BSTFA) (60 µL) was added. After reaction for 50 min in a water bath at 60 °C, the vial was taken out, cooled to room temperature and then analyzed by GC-MS directly. The Cambridge pad which collected the smoke particulate phase was transferred to the sample bottle, and dichloromethane (6 mL) was added containing 50 µg/mL IS. The samples were ultrasonically extracted at room temperature for 20 min, and supernatant (1 mL) was taken which was filtered with microporous membrane (0.45 µm) then transferred into the chromatogram vial with addition of BSTFA (60 µL) and reacted for 50 min in a water bath at 60 °C. Finally, the vial was taken out and cooled to room temperature then analyzed as before. Each cigarette sample was smoked and analysed in 3 repetitions, and the results were presented as 3 replicates.

GC-MS (Agilent 7890) conditions:

- 60 m × 0.25 mm i.d. × 0.25 µm;
- injector and detector temperature: 250 °C;
- split ratio: 10:1;
- injection volume: 1 µl;
- programming temperature: 40 °C

This was kept for 3 min, then heated 4 °C/min to 280 °C and maintained for 40 min.

- transmission line temperature: 28 °C;
- ion source temperature: 280 °C;
- ionization method: EI;
- ionization energy: 70 eV;

Monitoring modes, full scan mode and selected ion scan mode was used. The retention time and monitor ions of 30 organic acids are shown in Table 2.

### *Determination of yields of routine smoke components*

According to the corresponding smoking regimes the mainstream cigarette smoke yields of TPM, nicotine, NFDPM, carbon monoxide and water were determined after smoking cigarettes on a Cerulean SM450 smoking machine.

### *Filter retention and distribution patterns of organic acids*

The methods for analyzing the retention and distribution patterns of organic acids in the filter were similar to those previously reported (23). A schematic illustration and the parameters of how the filters were cut are shown in Figure 1.

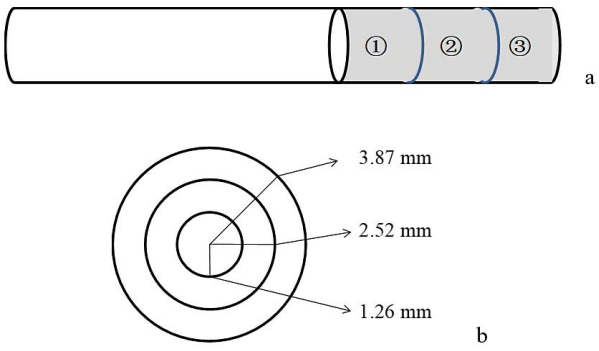


**Figure 1. Schematic illustration of filter transverse cuts (a) and concentric longitudinal cuts (b).**

**Table 2. The retention time, monitor ions, calibration curves, correlation coefficients, limits of detection (LOD), limits of quantification (LOQ), recovery and relative standard deviations (RSDs) of organic acids (n = 6)**.

| Components | Retention time (min) | Monitor ions (m/z) | Calibration curves | Correlation coefficients | LODs (s/n=3) (μg/cig) | LOQs (s/n=10) (μg/cig) | Recovery (%) | RSDs (%) |
|---|---|---|---|---|---|---|---|---|
| Formic acid | 5.9 | 103 [a] / 75 | y = 1.261x + 0.0263 | 0.9998 | 0.72 | 2.4 | 75.34 | 8.58 |
| Acetic acid | 7.81 | 117 [a] / 75 | y = 1.112x + 0.0137 | 0.9998 | 0.90 | 3.0 | 78.53 | 6.24 |
| Propionic acid | 10.69 | 131 [a] / 75 | y = 0.983x + 0.0026 | 0.9998 | 0.18 | 0.6 | 82.26 | 4.47 |
| Butyric acid | 14.04 | 145 [a] / 75 | y = 0.948x + 0.0027 | 0.9999 | 0.09 | 0.3 | 81.33 | 4.03 |
| 2-Methylbutyric acid | 15.62 | 159 [a] / 117 | y = 0.760x + 0.0011 | 0.9998 | 0.18 | 0.6 | 83.35 | 3.57 |
| 3-Methylbutyric acid | 16.04 | 159 [a] / 117 | y = 0.887x + 0.0005 | 0.9999 | 0.18 | 0.6 | 82.24 | 3.68 |
| *N*-Valeric acid | 17.89 | 159 [a] / 117 | y = 0.874x – 0.0004 | 0.9997 | 0.18 | 0.6 | 86.68 | 3.55 |
| 3-Methylvaleric acid | 20.11 | 159 [a] / 117 | y = 0.051x – 0.0002 | 0.9998 | 0.21 | 0.7 | 88.62 | 3.34 |
| 4-Methylvaleric acid | 20.34 | 173 [a] / 132 | y = 0.708x –0.0048 | 0.9997 | 0.21 | 0.7 | 89.35 | 3.29 |
| Lactic acid | 21.41 | 147 [a] / 117 | y = 1.001x + 0.0029 | 0.9999 | 0.15 | 0.5 | 87.24 | 4.24 |
| Hexanoic acid | 21.71 | 173 / 117 | y = 0.822x – 0.0011 | 0.9996 | 0.18 | 0.6 | 86.32 | 4.33 |
| IS | 23.57 | 171 [a] / 75 | | | | | | |
| 2-Furoic acid | 24.15 | 125 [a] / 169 | y = 1.327x – 0.0089 | 0.9993 | 0.12 | 0.4 | 83.37 | 4.56 |
| Heptanoic acid | 25.39 | 117 [a] / 75 | y = 0.435x – 0.0028 | 0.9994 | 0.18 | 0.6 | 84.56 | 3.28 |
| Malonic acid | 26.78 | 147 [a] / 73 | y = 2.860x – 0.2947 | 0.9991 | 0.09 | 0.3 | 87.25 | 3.39 |
| Benzoic acid | 28.33 | 179 [a] / 105 | y = 1.379x – 0.0088 | 0.9994 | 0.12 | 0.4 | 90.36 | 3.87 |
| Octanoic acid | 28.87 | 117 [a] / 75 | y = 0.494x – 0.0043 | 0.9998 | 0.18 | 0.6 | 93.37 | 3.61 |
| Succinic acid | 30.72 | 147 [a] / 73 | y = 1.031x – 0.0254 | 0.9999 | 0.09 | 0.3 | 91.25 | 2.36 |
| Nonanoic acid | 32.18 | 215 [a] / 117 | y = 0.712x – 0.0115 | 0.9998 | 0.18 | 0.6 | 92.23 | 3.25 |
| 2-Decenoic acid | 35.31 | 229 [a] / 117 | y = 0.652x – 0.0161 | 0.9997 | 0.18 | 0.6 | 91.69 | 3.27 |
| Malic acid | 36.64 | 147 [a] / 233 | y = 1.065x – 0.0753 | 0.9995 | 0.15 | 0.5 | 92.72 | 3.59 |
| Lauric acid | 41.01 | 117 [a] / 258 | y = 0.533x – 0.0323 | 0.9994 | 0.18 | 0.6 | 91.29 | 2.63 |
| Tridecanoic acid | 43.81 | 117 [a] / 272 | y = 0.515x – 0.0366 | 0.9996 | 0.18 | 0.6 | 89.87 | 3.19 |
| Myristic acid | 46.38 | 117 [a] / 286 | y = 0.022x – 0.0037 | 0.9991 | 0.12 | 0.4 | 88.53 | 3.28 |
| Pentadecanoic acid | 48.84 | 117 [a] / 132 | y = 0.512x – 0.0447 | 0.9998 | 0.18 | 0.6 | 88.86 | 3.62 |
| Palmitic acid | 51.18 | 117 [a] / 132 | y = 0.513x – 0.0425 | 0.9993 | 0.18 | 0.6 | 86.24 | 1.94 |
| Linoleic acid | 54.96 | 338 [a] / 263 | y = 0.049x – 0.0114 | 0.9990 | 0.42 | 1.4 | 90.39 | 3.45 |
| Oleic acid | 55.07 | 340 [a] / 117 | y = 0.148x – 0.0221 | 0.9996 | 0.39 | 1.3 | 88.76 | 3.13 |
| Linolenic acid | 55.14 | 336 [a] / 108 | y = 0.026x – 0.0064 | 0.9992 | 0.42 | 1.4 | 89.35 | 3.26 |
| Stearic acid | 55.59 | 342 [a] / 117 | y = 0.084x – 0.0113 | 0.9995 | 0.18 | 0.6 | 84.24 | 2.07 |

[a] ions for quantitation
s/n: signal-to-noise

Since the CA filter contains a small amount of residual acetic acid, it needed to be run as a blank and deducted when determining the amount of organic acid retention in the filter. The calibrated organic acid concentrations in different filter segments were processed by interpolation and polynomial fitting, and filter retention distributions of organic acids were obtained.

## RESULTS AND DISCUSSION

### *Calibration curves, LODs, LOQs, recovery and RSDs*

A series of standard mixture solutions of 30 organic acids were prepared. After these samples were derivatized with BSTFA, they were analyzed by GC-MS. The results showed that the linear correlation coefficients of all 30 organic acids were in the range of 0.9990–0.9999 (Table 2).

The chromatograms in selected ion monitoring (SIM) mode of the organic acids standard mixture solution are shown in Figure 2. An aliquot (1 mL) was taken of the lowest concentration organic acid standard solution for derivatization across 10 replicates to determine the standard deviation (SD). The limit of detection (LOD) and limit of quantification (LOQ) of the method was equal to 3 times SD and 10 times SD, respectively, and correspondingly the content of organic acids in cigarette smoke could be calculated. A known amount of organic acids standard mixture solution was added to the TPM samples, and the treatment process and quantitative analysis were performed in parallel and repeated 6 times.

The recovery was calculated based on the measurement results. The results in Table 2 show that the recovery of all acids was above 80% except for formic and acetic acids. This might result from the partial volatilization of formic acid and acetic acid during the treatment process. For establishing the relative standard deviations (RSDs), the same cigarette sample was tested 6 times under the same conditions. The results in Table 2 show that the RSDs of the 30 organic acids are all less than 8.6%, indicating that such a method has good repeatability and is suitable for the quantitative analysis of organic acids in cigarette smoke.

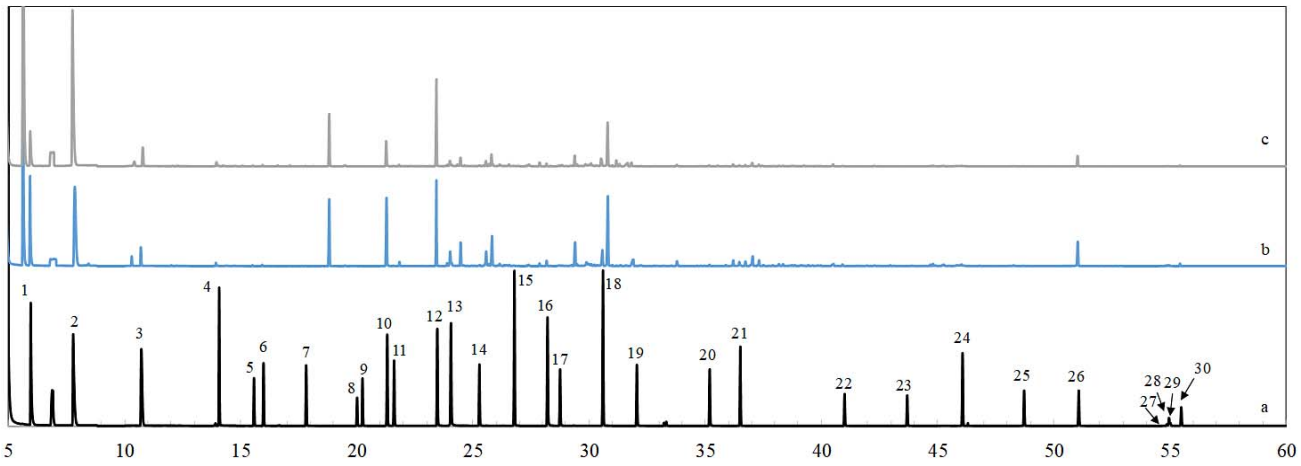


**Figure 2. Total ion chromatograms (SIM mode) of a standard organic acids mixture solution (a), TPM samples (b) and retention in filtration (c).** 1 = formic acid; 2 = acetic acid; 3 = propionic acid; 4 = butyric acid; 5 = 2-methylbutyric acid; 6 = 3-methylbutyric acid; 7 = *N*-valeric acid; 8 = 3-methylvaleric acid; 9 = 4-methylvaleric acid; 10 = lactic acid; 11 = caproic acid; 12 = IS; 13 = 2-furoic acid; 14 = heptanoic acid; 15 = malonic acid; 16 = benzoic acid; 17 = octanoic acid; 18 = succinic acid; 19 = nonanoic acid; 20 = 2-decenoic acid; 21 = malic acid; 22 = lauric acid; 23 = tridecanoic acid; 24 = myristic acid; 25 = pentadecanoic acid; 26 = palmitic acid; 27 = linoleic acid; 28 = oleic acid; 29 = linolenic acid; 30 = stearic acid. (27, 28, 29 separated by using respective monitor ions 338, 340, 336)

*The effect of filter ventilation levels on yields of organic acids in cigarette smoke under the ISO smoking regime and a comparison of the yields between ISO and HCI smoking regimes*

Table 3 shows the gas phase yields of organic acids at different filter ventilation levels under ISO and HCI smoking regimes. Only two organic acids were detected in the gas phase of mainstream cigarette smoke (i.e., formic acid and acetic acid). Compared with their yields in particulate phase, they accounted for only a small proportion (5%) of the total. By increasing ventilation of the filter, their yields gradually decreased. When the filter ventilation was 40%, the drop of formic acid was 23%, and the drop of acetic acid was 24%. Comparing with ISO smoking regime (0% ventilation), under HCI smoking regime (ventilation blocked), the yield of formic acid was increased by 47%, and the yield of acetic acid was increased by 44%.

Table 4 lists the yields and reduction ratios of the organic acids in cigarette smoke at five different filter ventilation levels under the ISO smoking regime, and with blocked-ventilation under HCI smoking regime.

It was found that the yields of formic acid, acetic acid, lactic acid, palmitic acid, linoleic acid and linolenic acid were all higher than 49 µg/cig, and the other organic acids were lower than 25 µg/cig when the filter was not ventilated. However, when the yield of carboxyl substances was calculated, the yields of formic acid, acetic acid and lactic acid were 1.07, 2.03, 0.78 mol/cig, respectively, and the other organic acids were lower than 0.35 mol/cig. Since the other organic acids have longer carbon chains and weaker acidity, these organic acids with larger amounts of carboxyl substances have a greater influence on the acid-base equilibrium of the cigarette smoke. When the filter ventilation was increased, the yields of all organic acids decreased, but in different proportions. The organic acids with lower BP and lower MW were more affected by the increase of ventilation, e.g., when the ventilation was 40%, the reduction ratio of formic acid was 58% and the reduction ratio of acetic acid was 57%. The organic acids with higher BP and higher MW were less affected by the increase of ventilation, e.g., BP of oleic acid and stearic acid were 360 °C and 361 °C, and the MWs were 282 amu (atomic mass unit) and 284 amu, respectively, which were relatively higher than those of the other organic acids. The reduction ratios for these two organic acids were 19% and 19% at 40% ventilation, which is lower than that of the other organic acids. These results are similar to those previously reported (20–21). Compared with the ISO

**Table 3. The yields of organic acids in the gas phase at different filter ventilation levels under the ISO smoking regime and the HCI smoking regime with blocked ventilation.**

| Compound | ISO | | | | | | | | | HCI | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | Ventilation 0% | Ventilation 10% | | Ventilation 20% | | Ventilation 30% | | Ventilation 40% | | Blocked | |
| | Delivery (µg/cig) | Delivery (µg/cig) | Reduction | Delivery (µg/cig) | Reduction | Delivery (µg/cig) | Reduction | Delivery µg/cig) | Reduction | Delivery (µg/cig) | Increase |
| Formic acid | 2.85 | 2.70 | 5.26% | 2.49 | 12.63% | 2.35 | 17.54% | 2.18 | 23.51% | 4.21 | 47.72% |
| Acetic acid | 6.37 | 6.07 | 4.71% | 5.64 | 11.46% | 5.38 | 15.54% | 4.82 | 24.33% | 9.22 | 44.74% |

**Table 4. The yields of organic acids in cigarette smoke (gas phase and TPM) at different filter ventilation levels under the ISO smoking regime and the HCI smoking regime with blocked ventilation.**

| Components | ISO | | | | | | | | | | HCI | | BP (°C) | MW |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | Ventilation 0% | | Ventilation 10% | | Ventilation 20% | | Ventilation 30% | | Ventilation 40% | | Blocked | | | |
| | Delivery (μg/cig) | ACS [a] (mol) | Delivery (μg/cig) | Reduction (%) | Delivery (μg/cig.) | Reduction (%) | Delivery (μg/cig) | Reduction (%) | Delivery (μg/cig) | Reduction (%) | Delivery (μg/cig) | Increase (%) | | |
| Formic acid | 49.12 | 1.07 | 40.17 | 18..2 | 34.83 | 29.1 | 27.51 | 44.0 | 20.52 | 58.2 | 136.96 | 117.7 | 101 | 46 |
| Acetic acid | 121.91 | 2.03 | 102.36 | 16.0 | 87.37 | 28.3 | 65.66 | 46.1 | 52.80 | 56.7 | 288.29 | 120.1 | 117 | 60 |
| Propionic acid | 9.71 | 0.13 | 8.24 | 15.2 | 7.08 | 27.1 | 5.93 | 38.9 | 4.47 | 54.0 | 28.92 | 197.8 | 141 | 74 |
| Butyric acid | 1.24 | 0.01 | 1.06 | 14.6 | 0.92 | 25.9 | 0.80 | 35.6 | 0.63 | 49.2 | 4.49 | 261.7 | 163 | 88 |
| 2-Methylbutyric acid | 1.77 | 0.02 | 1.56 | 12.1 | 1.38 | 22.2 | 1.24 | 30.1 | 1.01 | 43.1 | 3.36 | 89.1 | 176 | 102 |
| *N*-Valeric acid | 0.83 | 0.01 | 0.73 | 11.8 | 0.66 | 20.2 | 0.60 | 27.5 | 0.51 | 38.4 | 1.44 | 74.2 | 185 | 102 |
| Lactic acid | 70.05 | 0.78 | 59.34 | 15.3 | 50.08 | 28.5 | 42.54 | 39.3 | 31.97 | 54.4 | 142.57 | 103.5 | 122 | 90 |
| Hexanoic acid | 1.09 | 0.01 | 0.98 | 10.5 | 0.92 | 15.9 | 0.89 | 18.7 | 0.74 | 32.4 | 1.62 | 48.1 | 202 | 116 |
| 2-Furoic acid | 12.95 | 0.12 | 11.73 | 9.4 | 10.90 | 15.8 | 9.59 | 25.9 | 8.86 | 31.6 | 26.95 | 108.2 | 230 | 112 |
| Heptanoic acid | 3.53 | 0.03 | 3.16 | 10.5 | 2.97 | 15.9 | 2.76 | 21.8 | 2.43 | 31.2 | 5.28 | 49.6 | 223 | 130 |
| Malonic acid | 2.16 | 0.04 | 1.85 | 14.4 | 1.66 | 23.1 | 1.53 | 29.2 | 1.42 | 34.3 | 4.24 | 96.3 | 140 | 104 |
| Benzoic acid | 4.99 | 0.04 | 4.52 | 9.4 | 4.25 | 14.8 | 3.92 | 21.4 | 3.54 | 29.1 | 9.43 | 88.9 | 249 | 122 |
| Octanoic acid | 1.21 | 0.01 | 1.11 | 8.3 | 1.05 | 13.2 | 0.95 | 21.5 | 0.86 | 28.9 | 2.23 | 84.3 | 240 | 144 |
| Succinic acid | 0.97 | 0.02 | 0.88 | 9.3 | 0.84 | 13.4 | 0.77 | 20.6 | 0.72 | 25.8 | 1.35 | 39.2 | 236 | 118 |
| Nonanoic acid | 5.28 | 0.03 | 4.78 | 9.5 | 4.53 | 14.2 | 4.21 | 20.3 | 4.01 | 24.1 | 9.12 | 72.7 | 254 | 158 |
| 2-Decenoic acid | 2.40 | 0.01 | 2.18 | 9.2 | 2.04 | 15.0 | 1.93 | 19.6 | 1.78 | 25.8 | 3.45 | 43.8 | 270 | 172 |
| Malic acid | 5.18 | 0.08 | 4.68 | 9.7 | 4.33 | 16.4 | 4.09 | 21.0 | 3.98 | 23.2 | 8.64 | 66.8 | 306 | 134 |
| Lauric acid | 1.13 | 0.01 | 1.02 | 9.7 | 0.97 | 14.2 | 0.91 | 19.5 | 0.89 | 21.2 | 2.12 | 87.6 | 299 | 200 |
| Tridecanoic acid | 2.98 | 0.01 | 2.71 | 9.1 | 2.58 | 13.4 | 2.41 | 19.1 | 2.31 | 22.5 | 4.67 | 56.7 | 236 | 214 |
| Myristic acid | 2.56 | 0.01 | 2.33 | 9.0 | 2.25 | 12.1 | 2.12 | 17.2 | 2.04 | 20.3 | 4.25 | 66.0 | 250 | 228 |
| Pentadecanoic acid | 3.92 | 0.02 | 3.59 | 8.4 | 3.42 | 12.8 | 3.25 | 17.1 | 3.12 | 20.4 | 6.26 | 59.7 | 257 | 242 |
| Palmitic acid | 86.94 | 0.34 | 81.34 | 6.4 | 76.23 | 12.3 | 71.89 | 17.3 | 70.51 | 18.9 | 95.33 | 9.6 | 351 | 256 |
| Linoleic acid | 64.07 | 0.23 | 59.71 | 6.8 | 56.39 | 12.0 | 52.78 | 17.6 | 51.61 | 19.4 | 74.59 | 16.4 | 230 | 280 |
| Oleic acid | 13.65 | 0.05 | 12.79 | 6.3 | 12.07 | 11.6 | 11.28 | 17.4 | 11.01 | 19.3 | 18.30 | 34.0 | 360 | 282 |
| Linolenic acid | 97.53 | 0.35 | 89.39 | 8.3 | 86.75 | 11.0 | 80.12 | 17.8 | 78.53 | 19.5 | 128.06 | 31.3 | 230 | 278 |
| Stearic acid | 24.14 | 0.08 | 22.85 | 5.3 | 21.62 | 10.4 | 19.98 | 17.2 | 19.56 | 19.0 | 28.24 | 17.0 | 361 | 284 |
| Total | 591.31 | 5.46 | 525.06 | 11.2 | 478.09 | 19.1 | 419.66 | 29.0 | 379.82 | 35.8 | 990.13 | 67.4 | | |

[a] Amount of Carboxyl Substance (ACS) = Organic acid delivery / Molecular weight × Number of carboxyl groups

smoking regime, the puff volume of the HCI smoking regime is increased, puff interval is shortened, and the yields of organic acids is increased, but the extent of increase of different organic acids is different. The yield of formic acid was increased by 117%, and the yield of palmitic acid was only increased by 10%. In the HCI smoking regime the ventilation holes were blocked, thus no conclusions regarding the effect of filter ventilation on the yields of organic acids could be made.

*The effects of filter ventilation levels on yields of routine smoke components under the ISO smoking regime and a comparison of the yields between ISO and HCI smoking regimes*

The yields of routine smoke components at different filter ventilation levels under the ISO smoking regime and a comparison of the yields between ISO and HCI smoking regimes are shown in Table 5.

**Table 5. The yields of routine smoke components at different filter ventilation levels under the ISO smoking regime and the HCI smoking regime with blocked ventilation.**

| Compound | ISO | | | | | | | | | HCI | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | Ventilation 0% | Ventilation 10% | | Ventilation 20% | | Ventilation 30% | | Ventilation 40% | | Blocked | |
| | Delivery | Delivery | Reduction | Delivery | Reduction | Delivery | Reduction | Delivery | Reduction | Delivery | Increase |
| NFDPM (mg/cig) | 9.30 | 8.42 | 9.5% | 7.49 | 19.5% | 6.60 | 29.0% | 5.60 | 39.8% | 19.40 | 108.6% |
| TPM (mg/cig) | 11.39 | 10.21 | 10.4% | 8.97 | 21.3% | 7.85 | 31.1% | 6.57 | 42.3% | 29.40 | 158.1% |
| Nicotine (mg/cig) | 0.79 | 0.73 | 7.6% | 0.67 | 15.2% | 0.63 | 20.3% | 0.59 | 25.3% | 1.56 | 97.5% |
| Water (mg/cig) | 1.30 | 1.06 | 18.5% | 0.81 | 37.7% | 0.62 | 52.3% | 0.38 | 70.8% | 8.44 | 549.2% |
| CO (mg/cig) | 11.50 | 9.91 | 13.8% | 8.23 | 28.4% | 6.65 | 42.2% | 5.40 | 53.0% | 21.90 | 90.4% |
| Puff Number | 5.6 | 5.8 | 3.8% | 6.0 | 7.1% | 6.2 | 10.7% | 6.4 | 14.3% | 8.2 | 46.4% |

**Table 6. The filter retention of organic acids at different filter ventilation levels under the ISO smoking regime and the HCI smoking regime with blocked ventilation.**

| Components | ISO | | | | | | | | | HCI | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | Ventilation 0% | Ventilation 10% | | Ventilation 20% | | Ventilation 30% | | Ventilation 40% | | Blocked | |
| | Filter retention (μg/cig) | Filter retention (μg/cig) | Reduction (%) | Filter retention (μg/cig) | Reduction (%) | Filter retention (μg/cig) | Reduction (%) | Filter retention (μg/cig) | Reduction (%) | Filter retention (μg/cig) | Increase (%) |
| Formic acid | 61.86 | 56.41 | 8.8 | 48.35 | 21.8 | 42.36 | 31.5 | 37.59 | 39.2 | 147.79 | 138.9 |
| Acetic acid | 345.85 | 313.24 | 9.4 | 277.93 | 19.6 | 243.89 | 29.5 | 211.23 | 38.9 | 653.11 | 117.8 |
| Propionic acid | 33.08 | 30.38 | 8.2 | 27.48 | 16.9 | 24.61 | 25.6 | 21.84 | 34.0 | 74.24 | 124.4 |
| Butyric acid | 9.44 | 8.74 | 7.4 | 8.03 | 15.0 | 7.29 | 22.8 | 6.58 | 30.3 | 17.72 | 87.6 |
| 2-Methylbutyric acid | 3.25 | 3.12 | 4.0 | 2.76 | 15.1 | 2.56 | 21.2 | 2.34 | 27.9 | 7.13 | 119.3 |
| *N*-Valeric acid | 2.05 | 1.96 | 4.2 | 1.83 | 10.6 | 1.76 | 14.0 | 1.66 | 19.1 | 3.77 | 84.3 |
| Lactic acid | 63.99 | 57.23 | 10.6 | 50.29 | 21.4 | 45.87 | 28.3 | 39.84 | 37.7 | 103.38 | 61.5 |
| Hexanoic acid | 3.61 | 3.46 | 4.2 | 3.26 | 9.8 | 3.08 | 14.7 | 2.93 | 18.9 | 4.75 | 31.5 |
| 2-Furoic acid | 12.60 | 12.08 | 4.1 | 11.38 | 9.6 | 10.95 | 13.1 | 10.42 | 17.3 | 22.61 | 79.5 |
| Heptanoic acid | 4.51 | 4.31 | 4.4 | 4.08 | 9.5 | 3.88 | 14.0 | 3.68 | 18.4 | 7.63 | 69.2 |
| Malonic acid | 2.87 | 2.73 | 4.9 | 2.61 | 9.1 | 2.45 | 14.6 | 2.31 | 19.5 | 4.24 | 47.7 |
| Benzoic acid | 6.21 | 5.96 | 4.0 | 5.65 | 9.0 | 5.38 | 13.4 | 5.12 | 17.5 | 13.69 | 120.5 |
| Octanoic acid | 1.69 | 1.62 | 4.1 | 1.53 | 9.5 | 1.45 | 14.2 | 1.39 | 17.8 | 2.51 | 48.5 |
| Succinic acid | 1.54 | 1.48 | 3.9 | 1.41 | 8.4 | 1.37 | 11.0 | 1.28 | 16.9 | 2.14 | 39.0 |
| Nonanoic acid | 9.36 | 9.08 | 3.0 | 8.68 | 7.3 | 8.37 | 10.6 | 8.01 | 14.4 | 12.39 | 32.4 |
| 2-Decenoic acid | 3.53 | 3.42 | 3.1 | 3.27 | 7.4 | 3.19 | 9.6 | 3.05 | 13.6 | 5.58 | 58.1 |
| Malic acid | 8.47 | 8.24 | 2.7 | 7.93 | 6.4 | 7.69 | 9.2 | 7.38 | 12.9 | 13.77 | 62.6 |
| Lauric acid | 1.66 | 1.62 | 2.4 | 1.57 | 5.4 | 1.51 | 9.0 | 1.47 | 11.4 | 2.24 | 34.9 |
| Tridecanoic acid | 3.57 | 3.49 | 2.2 | 3.39 | 5.0 | 3.28 | 8.1 | 3.19 | 10.6 | 4.96 | 38.9 |
| Myristic acid | 4.12 | 4.02 | 2.4 | 3.94 | 4.4 | 3.80 | 7.8 | 3.74 | 9.2 | 6.53 | 58.5 |
| Pentadecanoic acid | 5.25 | 5.12 | 2.5 | 5.05 | 3.8 | 4.91 | 6.5 | 4.81 | 8.4 | 7.98 | 52.0 |
| Palmitic acid | 95.01 | 93.64 | 1.4 | 91.68 | 3.5 | 90.36 | 4.9 | 88.81 | 6.5 | 117.94 | 24.1 |
| Linoleic acid | 91.87 | 90.39 | 1.6 | 88.82 | 3.3 | 87.35 | 4.9 | 85.89 | 6.5 | 103.57 | 12.7 |
| Oleic acid | 49.06 | 48.58 | 1.0 | 47.72 | 2.7 | 47.06 | 4.1 | 46.38 | 5.5 | 49.81 | 1.5 |
| Linolenic acid | 105.34 | 104.39 | 0.9 | 103.29 | 2.0 | 102.24 | 2.9 | 101.23 | 3.9 | 129.07 | 22.5 |
| Stearic acid | 55.26 | 54.93 | 0.6 | 54.51 | 1.4 | 54.13 | 2.0 | 53.78 | 2.7 | 88.32 | 5.5 |

With increasing filter ventilation, the yields of routine smoke components were all reduced. The reduction ratios of NFDPM and TPM are similar to the filter ventilation levels. The BP (247 °C) and MW (162 amu) of nicotine are obviously higher than those of water. When the filter ventilation was 40%, the reduction of nicotine was only 25% and the reduction ratio of water was higher than 70%. Compared with the ISO smoking regime, the yield of nicotine increased by 97% and the yield of water increased by 549% under HCI smoking regime. Compared with the ISO smoking regime, both the higher puff volume and puff frequency of the HCI regime resulted in significantly increased yields of water. The results were in accordance with the ISO technical report (24).

*The effects of filter ventilation levels on filter retention of organic acids under the ISO smoking regime and a comparison of the filter retention between ISO and HCI smoking regimes*

In Table 6, it can be seen that the amount of organic acids retained in the filter is higher than the yield in mainstream cigarette smoke. This might be because organic acids contain carboxyl groups and are easily retained on the surface of CA. When the filter ventilation is increased, the retention of organic acids also decreases, and the degree of reduction of different organic acids is also different. For example, when the filter ventilation was 40%, the reduction of formic acid in filter retention was 39%, and the reduction of palmitic acid was only 5%. This also indicates that the smoke retention is not only influenced by smoke dilution, but is also related to spatial distribution in the filter. Comparing the ISO and the HCI smoking regime, with the increase of puff volume, the retention of all organic acids increased, but also to different extents, e.g., the retention of formic acid increased by 138%, and the retention of oleic acid only increased by 1.5%. In addition, the retention of benzoic acid increased by 120% when switching from the ISO to the HCI smoking regime, and the retention of other organic acids with similar BP and MW did not increase as much. This might be due to the weak polarity of benzoic acid and strong interaction with CA.

*The effects of filter ventilation levels on retention of routine smoke components under the ISO smoking regime and a comparison of the retention between ISO and HCI smoking regimes*

Table 7 shows the filter retention of routine smoke components at different filter ventilation levels under the ISO smoking regime and a comparison between the ISO and the HCI smoking regimes. It could be found that more water

**Table 7. The filter retention of routine smoke components at different filter ventilation levels under the ISO smoking regime and the HCI smoking regime with blocked ventilation.**

| Components | ISO | | | | | | | | | HCI | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | Ventilation 0% | Ventilation 10% | | Ventilation 20% | | Ventilation 30% | | Ventilation 40% | | Blocked | |
| | Filter retention (mg/cig) | Filter retention (mg/cig) | Reduction (%) | Filter retention (mg/cig) | Reduction (%) | Filter retention mg/cig. | Reduction (%) | Filter retention (mg/cig) | Reduction (%) | Filter retention (mg/cig) | Increase (%) |
| Nicotine | 0.52 | 0.50 | 3.8 | 0.49 | 5.8 | 0.48 | 7.7 | 0.47 | 9.6 | 1.02 | 96.2 |
| Water | 2.35 | 1.82 | 22.6 | 1.34 | 43.0 | 0.81 | 65.5 | 0.46 | 80.4 | 21.52 | 815.7 |

was retained in the filter than the yield in mainstream cigarette smoke. That might be due to the fact that CA has a certain degree of water absorption. With the increase of filter ventilation, the nicotine and water content in the filter decreased, but the degrees were significantly different. Comparing cigarettes with 0% and 40% filter ventilation, the filter retention of nicotine was only reduced by 9.6%, and the filter retention of water was reduced by 80%. Comparing the results obtained with the ISO and the HCI smoking regimes, the filter retention of nicotine increased by 96% and of water increased by as much as 815%.

*The effect of filter ventilation levels on the spatial distribution of organic acids in the filter*

Figure 3 shows the patterns of filter retention for formic acid at different filter ventilation levels. For unventilated cigarettes, the radial distribution of formic acid is concentrated in the middle of the filter, while longitudinally the concentration at the mouth end is higher than at the tobacco rod end, owing to the low BP and low MW of formic acid. It could also be found that the filter ventilation resulted in a significant decline of formic acid concentration on the periphery part and mouth end of the filter. Compared with the ISO smoking regime, the HCI regime causes an increase in the retention of formic acid in the filter, and the retention distribution is also slightly different.

The retention spatial concentration distribution patterns of lactic acid at different filter ventilation levels under ISO and HCI smoking regime are displayed in Figure 4. The influence of filter ventilation on the retention and distribution of lactic acid is similar to that of formic acid. With the increase of ventilation, the distribution of lactic acid was also more concentrated in the middle of the filter. Compared with the ISO smoking regime, in the HCI

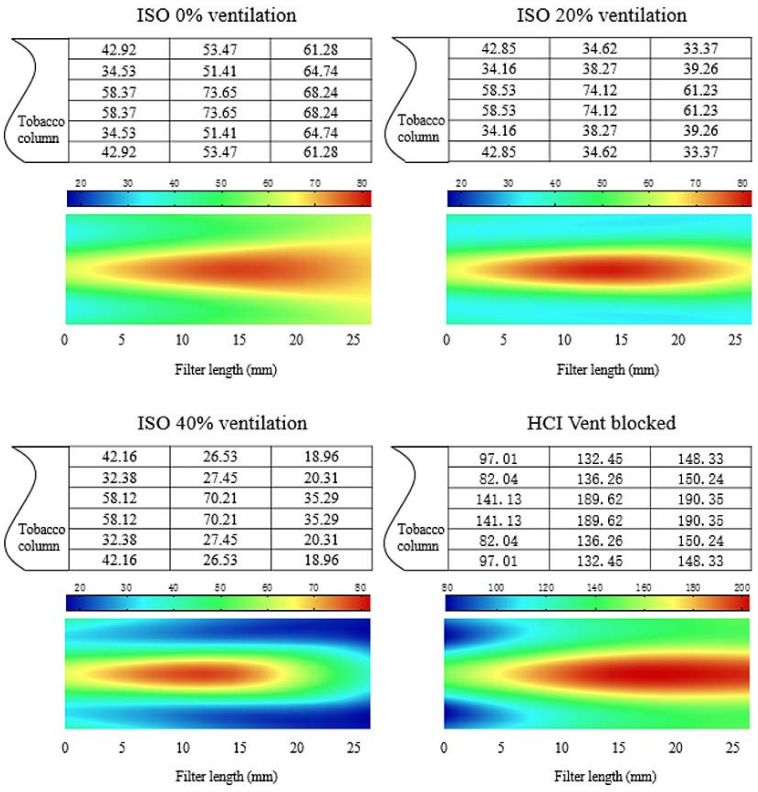


**Figure 3. Spatial distribution of formic acid in the filter at different filter ventilation levels under the ISO smoking regime and the HCI smoking regime with blocked ventilation (1000 × μg / mm$^3$).**

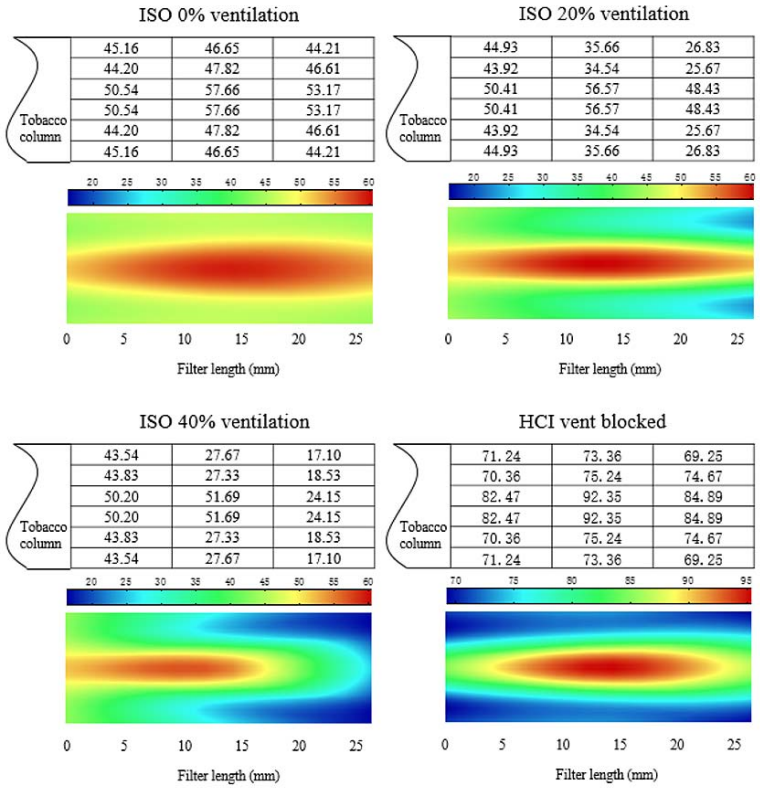


**Figure 4. Spatial distribution of lactic acid in the filter at different filter ventilation levels under the ISO smoking regime and the HCI smoking regime with blocked ventilation (1000 × μg / mm$^3$).**

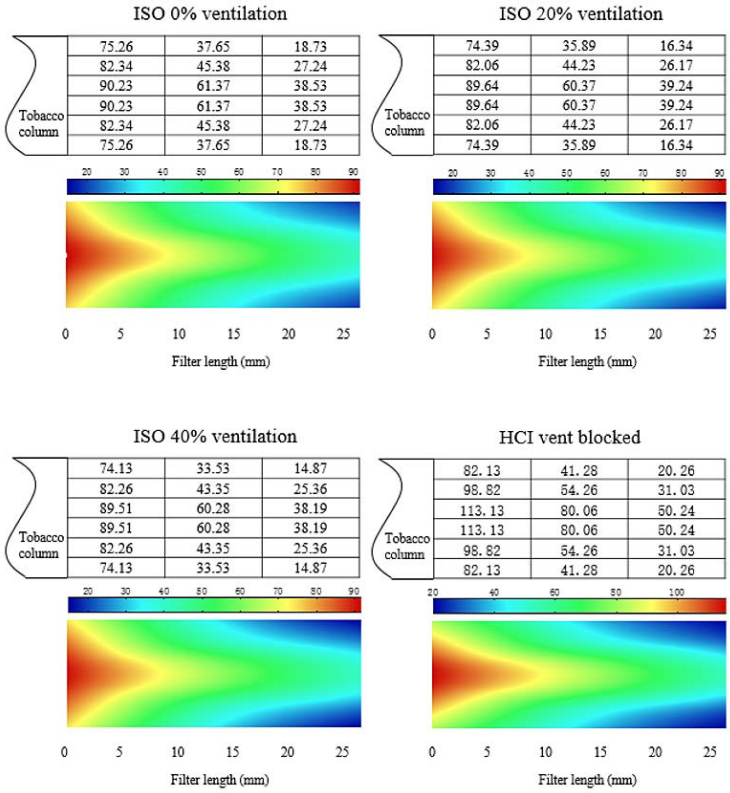


**Figure 5. Spatial distribution of palmitic acid in the filter at different filter ventilation levels under the ISO smoking regime and the HCI smoking regime with blocked ventilation (1000 × μg / mm$^3$).**

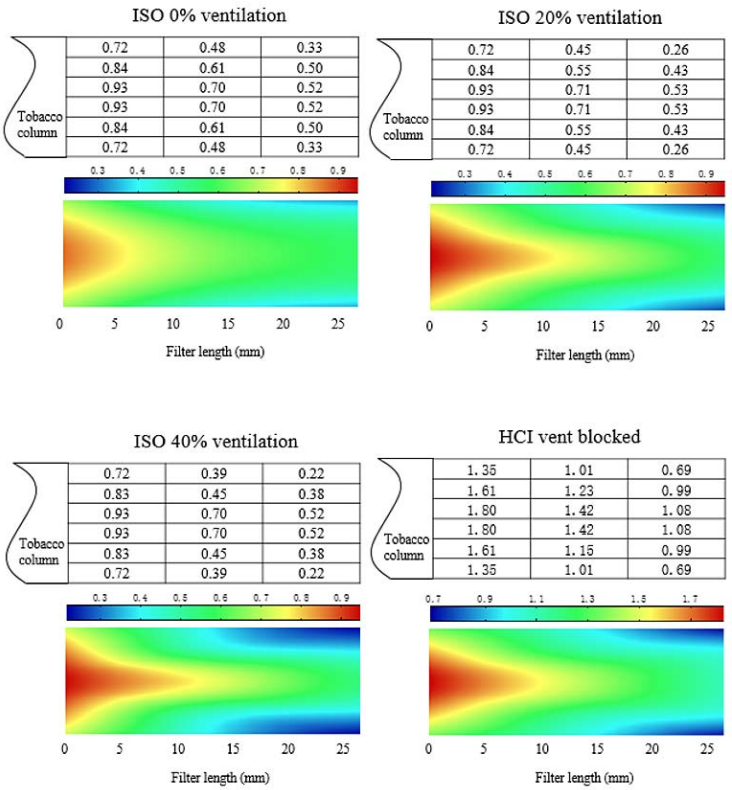


**Figure 6. Spatial distribution of nicotine in the filter at different filter ventilation levels under the ISO smoking regime and the HCI smoking regime with blocked ventilation (µg / mm$^3$).**

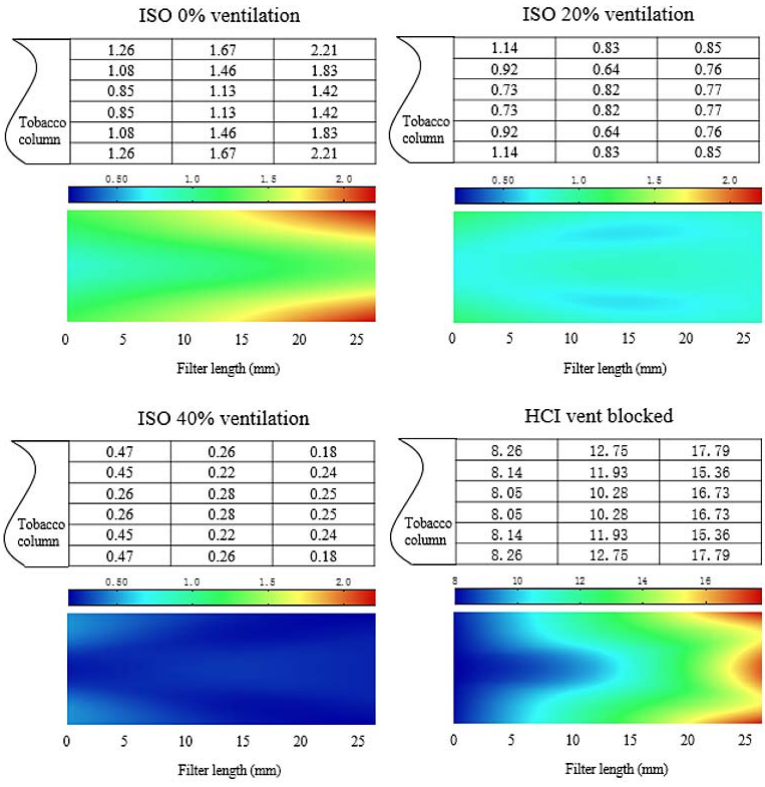


**Figure 7. Spatial distribution of water in the filter at different filter ventilation levels under the ISO smoking regime and the HCI smoking regime with blocked ventilation (µg / mm$^3$).**

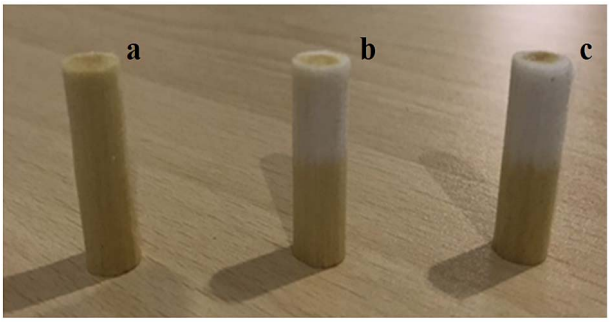


**Figure 8. Photograph of cigarette filters at different ventilation levels after smoking. (a) 0% ventilation, (b) 20% ventilation, and (c) 40% ventilation.**

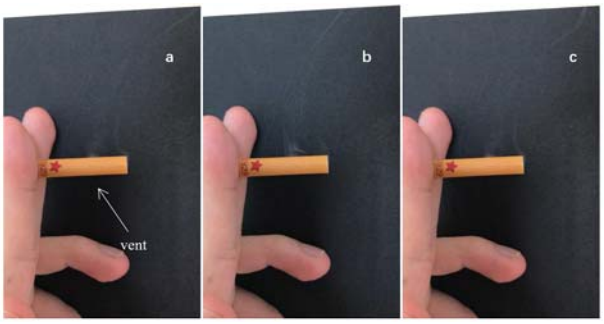


**Figure 9. Photographs of the 40% ventilated cigarette filter after smoking. (a) 0.2 second after a puff, (b) 0.5 second after a puff, and (c) 1 second after a puff.**

regime, the retention distribution of lactic acid was also more concentrated in the central area of the filter.

Palmitic acid has a higher BP and a larger MW than most other organic acids in this investigation, and its distribution in the filter is concentrated at the tobacco end of the filter. It can be seen from the Figure 5 that the filter ventilation had little effect on the retention and distribution of palmitic acid. The retention patterns of palmitic acid under ISO and HCI smoking regimes are also very similar.

As shown in Figures 6 and 7, the distribution of nicotine and water in the filter are significantly different. The distribution of nicotine is concentrated in the middle part and the tobacco rod end of the filter. Therefore, the filter ventilation and smoking regime have less influence on the distribution of nicotine in the filter.

Compared with nicotine, the BP and MW of water are lower. Therefore, the concentration of water in the lower temperature part at the mouth end and the peripheral part of the filter is higher. When the filter was ventilated, the concentration distribution of water was greatly affected. In addition, the HCI smoking resulted in higher water delivery and the concentration of water obviously increased at the mouth end of the filter. A photograph of cigarette filters at different filter ventilation levels after smoking is shown in Figure 8. It can be seen that filter ventilation has a significant effect on the retention of smoke. That is, the cigarette filter ventilation led to a reduction of the concentration of smoke aerosols in the peripheral part of the filter.

Photographs of the 40% ventilated cigarette filter after a puff of smoking are shown in Figure 9. It can be seen that the smoke diffused into the environment from the ventilation holes and the mouth end of the filter within 1 second after a puff of smoking. Therefore, the diffusion effect of chemical components with higher concentration distribution in these regions was also more significant. Under the HCI smoking regime, the ventilation holes are blocked, and the components with a high concentration distribution at the mouth end of the filter are more likely to diffuse into the mainstream smoke. As illustrated in Figure 10, a possible model for smoke diffusion in ventilated cigarette filter is proposed. When the filter is ventilated, the air compression and the smoke diffusion effect are generated around the vent holes.

In the ISO smoking regime, the puff profile is bell-shaped, and the smoke flow velocity is relatively fast for a short time (1–1.5 seconds). At this period, the air compression effect is obvious. With the flow velocity drops, the diffusion effect will become prominent. The diffusion of smoke will also continue for a period of time, about 2–3 seconds, after the end of each puff of smoking, due to the inertial effect of the smoke aerosol. Due to the smoke diffusion, the smoke concentration at the periphery part and smoking end of filter was greatly reduced.

As a result, the chemical components with a higher retention concentration in this part were more likely to diffuse into the environment, and finally caused an obvious decrease in the mainstream smoke. Owing to the air compression, the smoke concentration on the middle part was less affected. Therefore, the retention of these chemical components was not changed obviously, and the amount of these components entering the mainstream smoke was only affected by smoke dilution.

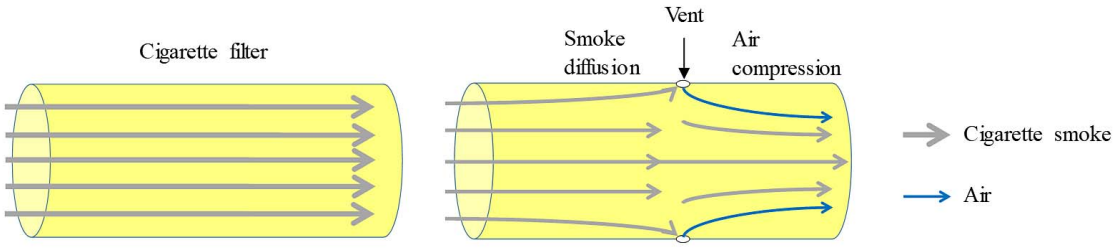


**Figure 10. A possible model for smoke diffusion in a ventilated cigarette filter.**

## CONCLUSION

The smoke dilution by filter ventilation has led to a decrease in yields of studied chemical components in cigarette smoke. The smoke diffusion and air compression produced by the filter ventilation has caused differences in the proportional reduction of these components. Compared with other organic acids in cigarette smoke, compounds such as formic acid, acetic acid, and lactic acid, have lower BP and lower MW, but the percentage of their carboxyl component is relatively larger, which might have had a greater impact on smoke pH. The concentration of these organic acids in the periphery of the filter was relatively high. When the filter was ventilated, these components in the mainstream cigarette smoke were greatly decreased. The distribution of palmitic acid and nicotine, which have higher BP and larger MW, was mainly concentrated in the middle part and the tobacco end of the filter, and is less affected by filter ventilation. These have led to changes in the chemical composition of mainstream cigarette smoke, which affected the sensory quality of the smoke. The HCI smoking regime did not reflect the benefits in yield reductions and sensory effects that filter ventilation had given to smoker, and it also led to different extents of increase in the release of different chemical components in the cigarette smoke. These results might be useful for the design and development of ventilated cigarettes and a better understanding of the yields and retention mechanisms of chemical components in ventilated cigarette filters.

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